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Indian Standard

ANIMAL FEEDING STUFFS — DETERMINATION OF MOISTURE AND OTHER VOLATILE MATTER CONTENT

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Price Group 4

Livestock Feeds, Equipment and Systems Sectional Committee, FAD 5

NATIONAL FOREWORD

This Indian Standard which is identical with ISO 6496 : 1999 'Animal feeding stuffs — Determination of moisture and other volatile matter content' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Livestock Feeds, Equipment and Systems Sectional Committee and approval of the Food and Agriculture Division Council.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker in the International Standards, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard, which is to be substituted in its place, is listed below along with its degree of equivalence for the edition indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 6498 : 1998 Animal feeding stuffs — Preparation of test samples	IS 14831 : 2000 Animal feeding stuffs — Preparation of test samples	Identical

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

ANIMAL FEEDING STUFFS — DETERMINATION OF MOISTURE AND OTHER VOLATILE MATTER CONTENT

1 Scope

This International Standard specifies a method for the determination of the moisture and other volatile matter content of animal feeding stuffs.

The method is applicable to animal feeding stuffs with the exception of:

a) milk products;

b) mineral substances;

c) mixtures containing a considerable amount of milk products or mineral substances, for example milk replacers;

d) animal feeding stuffs containing humectants (e.g. propylene glycol);

e) the following simple animal feeding stuffs:

- animal and vegetable fats and oils (for which method A specified in ISO 662 [1] is applicable);
- oilseeds (for which a method is specified in ISO 665 [2]);
- oilseed residues (for which a method is specified in ISO 771 [3]);
- cereals, except maize, and cereal products (for which a method is specified in ISO 712 [4]);
- maize (for which the reference method specified in ISO 6540 [5] is applicable).

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, such publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6498, *Animal feeding stuffs — Preparation of test samples.*

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
moisture and other volatile matter content
mass fraction of substances lost on drying the sample by the procedure specified in this International Standard

NOTE The moisture and other volatile matter content is expressed as a mass fraction in percent [formerly given as % (*m*/*m*)].

4 Principle

The loss of mass of a test portion of the sample on drying is determined under specified conditions depending on the nature of the sample.

5 Apparatus and materials

Usual laboratory apparatus and materials and, in particular, the following.

5.1 **Analytical balance**, capable of weighing to the nearest 1 mg.

5.2 **Container**, of non-corrodible metal or glass, provided with a sufficiently tight-fitting lid, and having a surface allowing the test portion to be spread to about 0,3 g/cm^2.

5.3 **Electrically heated oven**, well ventilated, capable of being maintained at a temperature of 103 °C ± 2 °C.

5.4 **Electrically heated vacuum oven**, capable of being maintained at a temperature of 80 °C ± 2 °C, and capable of producing a pressure below 13 kPa.

It shall be provided with a thermostat and vacuum pump, and either a device for the introduction of dried air, or a device containing calcium oxide (CaO) as desiccant (300 g CaO for 20 samples).

5.5 **Desiccator**, provided with an efficient desiccant.

5.6 **Sand**, acid-washed.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 6497 [6].

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Store the sample in such a way that deterioration and change in composition are minimized.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 6498.

8 Procedure

8.1 Test portion

8.1.1 Feeding stuffs in liquid or paste form, and feeding stuffs predominantly composed of oils and fats

Place a thin layer of sand (5.6) and a glass rod into a container (5.2). Dry the container with its contents and lid in the oven (5.3) set at 103 °C for 30 min ± 1 min. Place the lid on the container, remove from the oven and allow to cool to ambient temperature in the desiccator (5.5). Weigh the container with its contents and lid to the nearest 1 mg.

Into the container, weigh, to the nearest 1 mg, about 10 g of the prepared test sample (clause 7). Using the glass rod, mix thoroughly with the sand. Leave the glass rod in the container. Proceed in accordance with 8.2.

8.1.2 Other feeding stuffs

Dry a container (5.2) with its lid in the oven (5.3) set at 103 °C for 30 min ± 1 min. Remove from the oven and allow to cool to ambient temperature in the desiccator (5.5). Weigh the container with its lid to the nearest 1 mg.

Into the container, weigh, to the nearest 1 mg, about 5 g of the prepared test sample (clause 7) and spread evenly.

8.2 Determination

Place the container, with its lid beneath or beside it, in the oven (5.3) set at 103 °C. It is recommended that not more than one container per litre of oven volume be placed in the oven.

Leave to dry for 4 h ± 0,1 h after the oven temperature has returned to 103 °C . Place the lid on the container, remove from the oven, allow to cool to ambient temperature in the desiccator (5.5) and weigh to the nearest 1 mg.

Feeding stuffs consisting mainly of oils or fats shall be dried for another 30 min ± 1 min in the oven (5.3) set at 103 °C. The change in mass between the two weighings shall not exceed 0,1 % of the mass of the test portion. If the change in mass is more than 0,1 % of the mass of the test portion, discard the result and repeat the procedure. If the change in mass is again more than 0,1 % of the mass of the test portion, proceed in accordance with 8.3.

8.3 Check test

In order to check whether during drying of the test portion an unacceptable change in mass occurs as a result of chemical reactions (for example Maillard reactions), proceed as follows.

Dry the container and test portion again in the oven (5.3) set at 103 °C for 2 h ± 0,1 h. Allow to cool to ambient temperature in the desiccator (5.5) and weigh to the nearest 1 mg. If the change in mass during this second drying period is more than 0,2 % of the mass of the test portion, chemical reactions may have occurred. In this case, discard the result and apply the procedure specified in 8.4.

NOTE The criterion of 0,2 % of the mass of the test portion should not be confused with the repeatability limit of 0,2 % defined in 10.2. The latter concerns the absolute difference between two independent single test results obtained under repeatability conditions. The former is based on the difference between two weighings of the same test portion before and after an extra period of heating in order to check whether an unacceptable change in mass has occurred.

8.4 Samples giving unacceptable change in mass

Take the test portion in accordance with 8.1.

Place the container, with its lid beneath or beside it, in the vacuum oven (5.4) set at 80 °C. Reduce the pressure to about 13 kPa and dry the sample at this pressure, either while admitting dried air, or in the presence of the desiccant (see 5.4). In the latter case, disconnect the vacuum pump after the specified pressure has been reached and ensure that this pressure is maintained throughout the drying period. Heat the sample for 4 h ± 0,1 h after the oven temperature has returned to 80 °C. Return the oven pressure carefully to atmospheric pressure. Open the oven, immediately place the lid on the container, remove from the oven, allow to cool to ambient temperature in the desiccator (5.5) and weigh to the nearest 1 mg.

Dry for additional periods of 30 min ± 1 min in the vacuum oven set at 80 °C and weigh until the change in mass between two consecutive weighings does not exceed 0,2 % of the mass of the test portion.

8.5 Number of determinations

Carry out two determinations on test portions taken from the same test sample.

9 Expression of results

9.1 Determination without preliminary conditioning

Calculate the moisture and other volatile matter content of the test sample, w_1, in percent, by the equation:

$$w_1 = \frac{m_3 - (m_5 - m_4)}{m_3} \times 100\ \%$$

where

m_3 is the mass, in grams, of the test portion;

m_4 is the mass, in grams, of the container with lid including, if appropriate, sand and glass rod;

m_5 is the mass, in grams, of the container with lid and dried test portion including, if appropriate, sand and glass rod.

9.2 Determination with preliminary conditioning

NOTE For samples which are difficult to crush, see ISO 6498.

9.2.1 Samples with moisture content more than 17 % and fat content less than 120 g/kg, which need only preliminary drying

Calculate the moisture and other volatile matter content of the test sample, w_2, in percent, by the equation:

$$w_2 = \left[\frac{m_0 - m_1}{m_0} + \left(\frac{m_3 - (m_5 - m_4)}{m_3} \times \frac{m_1}{m_0}\right)\right] \times 100\ \%$$

where

m_0 is the mass, in grams, of the test sample;

m_1 is the mass, in grams, of the test sample after extraction and/or drying and conditioning in ambient air;

m_3 is the mass, in grams, of the test portion;

m_4 is the mass, in grams, of the container with lid including, if appropriate, sand and glass rod;

m_5 is the mass, in grams, of the container with lid and dried test portion including, if appropriate, sand and glass rod.

9.2.2 Samples with high fat content and low moisture content, which need only preliminary defatting, and samples with high fat content and high moisture content, which need preliminary drying followed by preliminary defatting

Calculate the moisture and other volatile matter content of the test sample, w_3, in percent, by the equation:

$$w_3 = \left[\frac{m_0 - m_1 - m_2}{m_0} + \left(\frac{m_3 - (m_5 - m_4)}{m_3} \times \frac{m_1}{m_0}\right)\right] \times 100\ \%$$

where m_2 is the mass, in grams, of the fat extracted from the test sample (see ISO 6498).

For the explanation of the other symbols, see 9.2.1.

9.3 Results

Take as the result the arithmetic mean of the two intermediate test results (8.5), provided that the absolute difference between the two results does not exceed 0,2 %. Repeat the procedure if the difference exceeds 0,2 %.

Express the result to the nearest 0,1 %.

10 Precision

10.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices others than those given.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases exceed the repeatability limit r given in or derived from Table 1.

Table 1 — Repeatability limit (r) and reproducibility limit (R)

Sample	Moisture and other volatile content %	 %	R %
Mixed feed	11,43	0,71	1,99
Mixed feed concentrate	10,20	0,55	1,57
Molassed feed	7,92	1,49	2,46
Dried grass	11,77	0,78	3,00
Beet pulp	86,05	0,95	3,50
Alfalfa (lucerne)	80,30	1,27	2,91

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases exceed the reproducibility limit R given in or derived from Table 1.

11 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the sampling method used, if known;
- the test method used, with reference to this International Standard;
- all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- the test result obtained, or the two test results obtained if the repeatability has been checked.

Annex A
(informative)

Results of interlaboratory test

An interlaboratory test was organized by ISO/TC 34/SC 10, *Animal feeding stuffs*, in 1996 and carried out in accordance with ISO 5725-2:1994 [8]. In the test 23 laboratories participated. Samples of mixed feed, mixed feed concentrate, molassed feed, dried grass, beet pulp and alfalfa (lucerne) were investigated.

Table A.1 — Statistical results of the interlaboratory test

Parameter	Sample[a]					
	1	2	3	4	5	6
Number of laboratories retained after eliminating outliers	23	23	19	23	23	23
Mean moisture and other volatile matter content, %	11,43	10,20	7,92	11,77	86,05	80,30
Repeatability standard deviation, s_r, %	0,253	0,195	0,533	0,28	0,34	0,454
Repeatability coefficient of variation, %	2,21	1,91	6,73	2,38	0,40	0,57
Repeatability limit r ($2,8 \times s_r$), %	0,71	0,55	1,49	0,78	0,95	1,27
Reproducibility standard deviation, s_R, %	0,71	0,562	0,878	1,07	1,25	1,04
Reproducibility coefficient of variation, %	6,22	5,51	11,09	9,09	1,45	1,30
Reproducibility limit, R ($2,8 \times s_R$), %	1,99	1,57	2,46	3,00	3,50	2,91

[a] Sample 1: mixed feed;
Sample 2: mixed feed concentrate;
Sample 3: molassed feed;
Sample 4: dried grass;
Sample 5: beet pulp;
Sample 6: alfalfa (lucerne).

Bibliography

[1] ISO 662, *Animal and vegetable fats and oils — Determination of moisture and volatile matter content.*

[2] ISO 665, *Oilseeds — Determination of moisture and volatile matter content.*

[3] ISO 771, *Oilseed residues — Determination of moisture and volatile matter content.*

[4] ISO 712, *Cereals and cereal products — Determination of moisture content (Routine reference method).*

[5] ISO 6540, *Maize — Determination of moisture content (on milled grains and on whole grains).*

[6] ISO 6497, *Animal feeding stuffs — Sampling.*

[7] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[8] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

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